REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

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1. REPORT DATE (DD-MM-YYYY) 15-08-2006	2. REPORT TYPE Final Technical	3. DATES COVERED (From - To) 01-05-2005 – 30-04-2006

4. TITLE AND SUBTITLE
(U) (DURIP 05) Laser-Based Optical System for Reactive Radical Concentration Measurements in Plasmas and Flames

5a. CONTRACT NUMBER

5b. GRANT NUMBER
FA9550-05-1-0320

5c. PROGRAM ELEMENT NUMBER
61103F

6. AUTHOR(S)

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5d. PROJECT NUMBER
5094

5e. TASK NUMBER
US

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

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Philadelphia PA 19104-2884

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)
AFOSR/NA
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10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S REPORT NUMBER(S)

AFRL-SR-AR-TR-06-0397

12. DISTRIBUTION / AVAILABILITY STATEMENT

Approved for public release; distribution is unlimited

13. SUPPLEMENTARY NOTES

14. ABSTRACT
Defense University Research Instrumentation Program (DURIP) funding was used to assemble a multi-purpose optical diagnostic system for in-situ radical concentration measurements in plasma and combustion systems. The system was configured to allow two-dimensional measurements (using a single wavelength) of important radical concentrations in stationary or time-averaged plasma and flame conditions, to make simultaneous nanosecond-resolved single point measurements at this wavelength, and to collect one-dimensional information about the spectral lines in rotational bands for the species of interest. The system consisted of a Nd:YAG pumped optical parametric oscillator (OPO), a Raman spectrometer, two intensified CCD cameras, and associated optics.

15. SUBJECT TERMS

Instrumentation, spectrometer, combustion, plasma, Raman, fluorescence, laser

16. SECURITY CLASSIFICATION OF:

a. REPORT	b. ABSTRACT	c. THIS PAGE	17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON Julian M. Tishkoff
Unclassified	Unclassified	Unclassified	UL	25	19b. TELEPHONE NUMBER (include area code) (703) 696-8478

Best Available Copy

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std. Z39.18

Laser-Based Optical System for Reactive Radical Concentration Measurements in Plasmas and Flames

Final Performance Report

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August 2006

20061016134

Introduction

Funds were requested to assemble a multi-purpose optical diagnostic system for in-situ radical concentration measurements in plasma and combustion systems. The system was configured to allow 2-D measurements (using a single wavelength) of important radical concentrations in stationary (or time-averaged) plasma or flame conditions, to make simultaneous nano-second resolved single point measurements at this wavelength, and, in an alternative arrangement, to collect 1-D spectra information for the spectral lines in rotational bands for the species of interest. The system consisted of a Nd:YAG pumped Optical Parametric Oscillator (OPO), a Raman Spectrometer, two Intensified CCD Cameras, and associated optics. The OPO system was already in place, as the result of DURIP-2001 program funding (contract DAAD19-01-1-0438); it can provide continuously tunable laser output from 400 nm to 1.7 μm, and it is configured for single point measurements of stable and transient chemical species. To achieve the additional capabilities described above, funding was awarded for the purchase of a Raman Spectrometer (TriVista, Princeton Instruments), two 18-mm Intensified CCD Cameras (PI-MAX2 – 512, Princeton Instruments), and some additional optics. This system was designed to enhance our diagnostics capabilities applicable for the study of plasma and combustion processes and contribute to our ongoing research programs, as well as, opening up new areas for future research.

Our primary interests in plasma and flame radical measurements include quantifying concentrations of the following radicals: HO_2, OH, O, CH, CN, and RO_2 and active molecular species, in particular, hydrogen peroxide (H_2O_2) and HCN. Measurements in Gliding Arc, Dielectric Barrier Discharge and Pulsed Corona Plasma systems and in flame and flow reactor systems are underway. To measure the low concentration of these radicals, we have used the Cavity Ringdown Laser Absorption Spectroscopy (CRLAS) technique and our modification which combines CRLAS with Magneto-Optical Rotation to enhance the selectivity of the measurement. The experimental data are of significant importance to development and testing of chemical kinetic models of processes, particularly in the low and intermediate temperature regimes.

The total requested budget was $209,800. The total received budget was $129,712. The received budget was used to purchase the Raman Spectrometer (TriVista, Princeton Instruments) and one 18-mm Intensified CCD Camera (see the attached quotation at the end of the document). The purchased equipment was installed in our laboratory and put into operation. The following paragraphs describe the experimental progress that has been achieved using this equipment. A list of publications and presentations using this data is included below.

As it is possible to see from the presented material, the purchased equipment is in active use and plays an important role in our investigations, including the military related projects

Projects and research tasks

In this chapter the projects, research tasks and obtained results are listed and described that include use of the developed spectroscopic system.

"Enhancement of Combustion and Flame Stabilization Using Transient Non-Equilibrium Plasma" (AFOSR, contract # FA9550-04-1-0038 via Princeton University)

In supersonic combustion, the flow speed is so high that there is only a very short residence time (less than 1 ms) for ignition, flame stabilization and reaction completion. In particular, the interest of hydrocarbon fuel due to its high density and endothermic decomposition properties results in a longer ignition and combustion time than that of hydrogen. Therefore, there is a crucial need to dramatically shorten the ignition time and to enhance the

combustion and flame stabilization. The subject of this project is investigation of the efficiency of unsteady non-equilibrium gliding arc in generation radicals and other chemical species for the enhancement of combustion. The gliding arc (GA) has the advantages of both equilibrium plasma and non-equilibrium plasma. It can operate at atmospheric pressure and simultaneously has high power and high electron density and electron temperature because of high reduced electric field (E/N). It also has high selectivity of plasma chemistry. We planed to combine the gliding arc with the counterflow flame burner to get a simplified flame geometry to study the fundamental properties of combustion and flame stabilization.

A novel magnetically stabilized gliding arc (MGA) (Fig. 1) reactor coupled with a counterflow burner was developed (Fig. 2) to study non-thermal plasma enhancement of ignition and extinction phenomena. The results showed that the new coupled plasma-flame system provides a well defined platform for understanding of the basic mechanism of the plasma-flame interaction. It was shown that with a plasma discharge of the air stream, up to a 220 percent increase in the extinction strain rate was possible at low power inputs for air and methane diluted with nitrogen. Measurements of temperature profiles via planar Rayleigh scattering thermometry and OH number density profiles via planar laser induced fluorescence (calibrated with absorption) were taken to quantify various effects. Detailed numerical simulations at elevated air temperatures and radical addition were performed for comparison with experimentally obtained results.
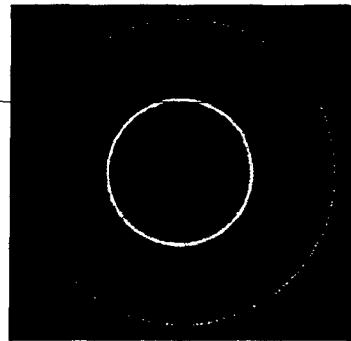
Fig. 1: Magnetic Gliding Arc
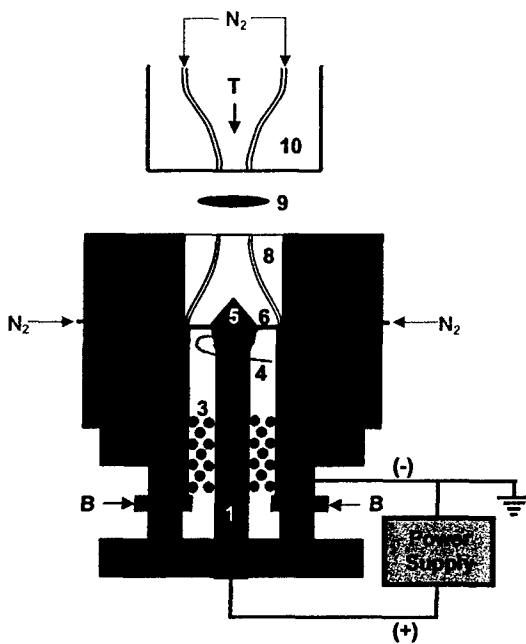
Results of the extinction experiments initially suggested that the enhancement effect was predominantly thermal for our particular setup of experiments. However, in ignition experiments specifically for hydrogen, temperature measurements conducted for hydrogen-air mixtures suggested the contribution of active species to justify the extent of enhancement effect. Further comparison with numerical simulations also provides an insight into the participation of species other than radicals in the enhancement effect.

Fig. 2: Counterflow burner with integrated plasma system. 1. cathode, 2. anode, 3. diffuser, 4. gliding arc initiation wire, 5. insulator, 6. plasma disk, 7. magnet, 8. converging nozzle with N_2 curtain, 9. flat diffusion flame, 10. water cooled converging nozzle with N_2 curtain.

The flat GA has been well studied over the years for its transition from the thermal to non-thermal regime. However, the MGA is a relatively new discharge which needs to be studied in order to characterize its non-equilibrium nature. Optical diagnostic techniques were employed in order to obtain average rotational and vibrational temperatures in the MGA with emission spectra obtained from OH and N_2 species from the discharge were used. We have developed a plasma diagnostic system based on optical emission spectroscopy from the air plasma. At the beginning an Acton Research SpectraPro 500i scanning monochromator was used, and then we also used Princeton Instruments Acton Research TriVista spectrometer with Intensified CCD

Camera. The entrance slit to the spectrometer was placed a few inches away from the discharge, facing the discharge. Spatially averaged emission spectra of the discharge were taken in a range of 200 nm – 450 nm. CCD camera was mounted onto the exit slit to digitally acquire the spectra. When the discharge was not in operation, a background noise spectrum was obtained. This was then subtracted from experimental data in order to improve the accuracy of the results. A low pressure mercury lamp was used to determine the slit (apparatus) function and calibrate the spectrometer.

For estimations of the current density, an imaging system was used and consisted of a Nikon D70 SLR camera, with a Sony make CCD type sensor. The sensor of size, 23.7mm x 15.5mm (Nikon DX), was used to capture images at a resolution of 3008 x 2000 pixels (~ 6 mega pixels). The shutter speeds available ranged from 30 seconds to 1/8000 seconds, which could be used to capture instantaneous arc images. These images were used to determine the approximate widths of the channels at various zones in the plasma discharge.

OH Spectrum for Rotational Temperature

A spatially averaged optical spectrum was obtained from the MGA plasma disk operating at approximately 32 mA, 37 Watt at normal pressure in air. The experimental spectrum, as seen in Fig. 3, had three major lines, G_0, G_1, and G_{REF} that were of the most interesting in the temperature range from 1000K – 4000K. This is because the reference peak, G_{REF} is the strongest group of unresolved lines and G_0 and G_1 are highly sensitive to temperature variation in this range. For temperatures above 4000K their sensitivity is relatively low.[26] Hence, the ratios of these peaks are best considered for diagnostic analysis. A theoretical spectrum was then generated by using the technique specified in Izarra[26] and Pellerin et al.[27] by collecting data pertaining to the OH spectrum as studied by Dieke & Crosswhite[28] in the paper of Izarra[26], wherein weak transitions were neglected and a set of delta functions corresponding to the strong transitions were generated from the relation below,

$$I_{nm} = I_{nm_{ref}} \frac{Z(T_{ref})}{Z(T)} e^{\frac{-E_n*(T_{ref}-T)}{(T*T_{ref})}}$$

Where, I_{nm} is the intensity of the OH transitions (nm) and occurs at a specific wavelength, E_n is the energy of the initial state and T_{ref} is the temperature at which the reference intensities $I_{nm_{ref}}$ were obtained. The parameters were taken from the fundamental data found in Izarra.[26] There, the ratio of the partition functions, $Z(T_{ref})/Z(T)$, was assumed to be unity. By using different values of temperature, T, the corresponding normalized intensities were computed. Delta functions corresponding to each transition were then convoluted with an apparatus function (i.e. impulse response of the optical device) obtained by employing sharp lines emitted by a low pressure mercury lamp. Figure 3 shows a theoretical plot for temperatures in 500K increments for the range of 1000K – 6000K and their comparison with those measured in experiments. The best fit of the ratios of G_0 and G_1 to G_{REF} yielded a rotational temperature of 2350K ± 150K, as shown in Fig. 4. The rotational temperature results obtained from the theoretical analysis above were compared with Spectrum Analyzer[29] software to identify peaks and compute rotational and vibrational temperatures. The results obtained from Spectrum Analyzer are more refined when larger numbers of correctly identified lines are used. The results obtained (Fig. 5) agreed with our calculations giving an average OH rotational temperature of ~ 2360K ± 400K. The vibrational temperature computed (Fig. 6) was found to be ~ 3500K ± 600K.
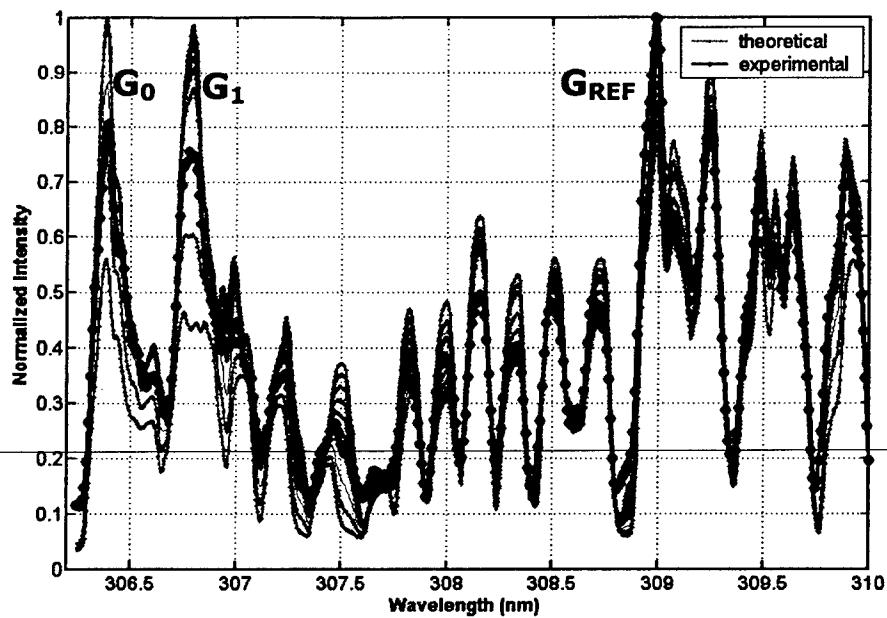
Fig. 3: The OH Spectrum obtained both experimentally (at ~ 80 Watt) and theoretically for different temperatures varying from 1000 K – 6000 K.
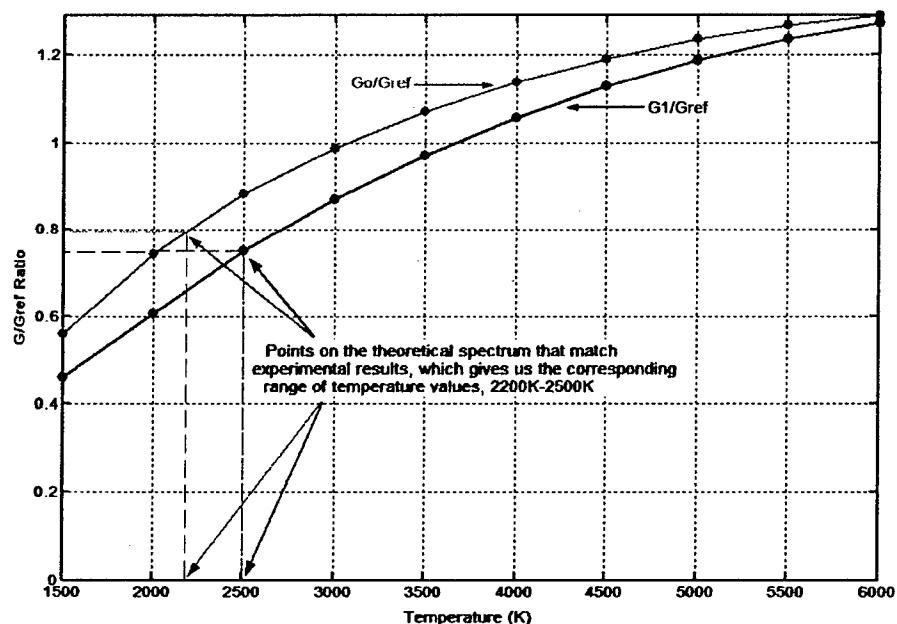

Fig. 4: The ratios of G_0/G_{REF} and G_1/G_{REF} peaks as a function of OH rotational temperature in the MGA. The comparison of the above theoretically generated plot with experimental result, yields a temperature of $2350 \pm 150K$
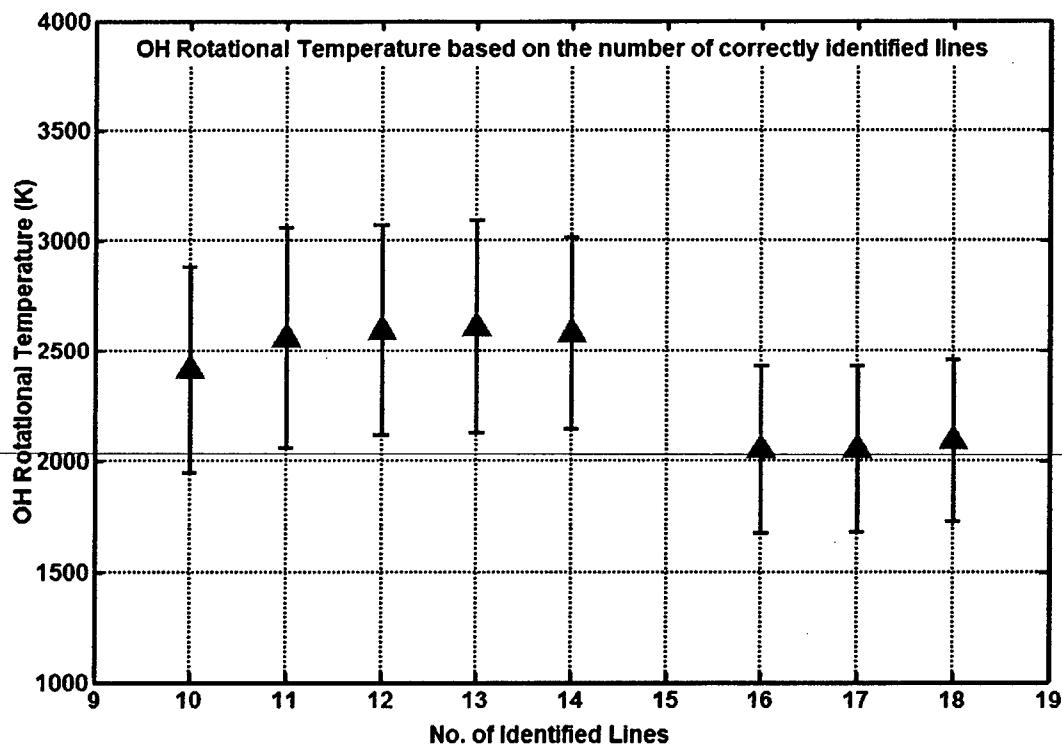
Fig. 5: Spectrum Analyzer OH Rotational Temperature.
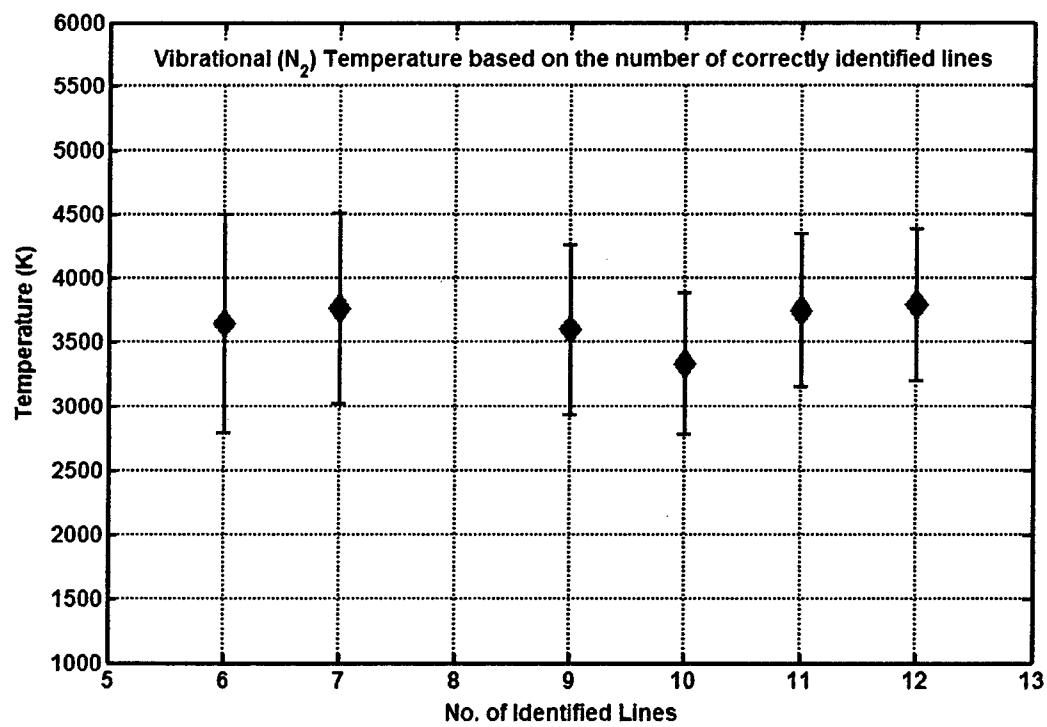
Fig. 6: Spectrum Analyzer N_2 Vibrational Temperature.

N₂ Spectrum for Rotational and Vibrational Temperature

A spatially averaged N_2 spectrum was obtained from the MGA plasma disk operating at approximately 37 Watts at normal pressure in air. Using the SpecAir[30] code, the experimental and theoretical plots were compared and using best fit analysis, the N_2 rotational temperature obtained was ~ 3000K. This result was in agreement with investigations reported earlier pertaining to gliding arcs.[31,32] Fig. 7 shows the fit of theoretical and experimental plots, with the best fit obtained for a vibrational temperature of ~ 4000K.
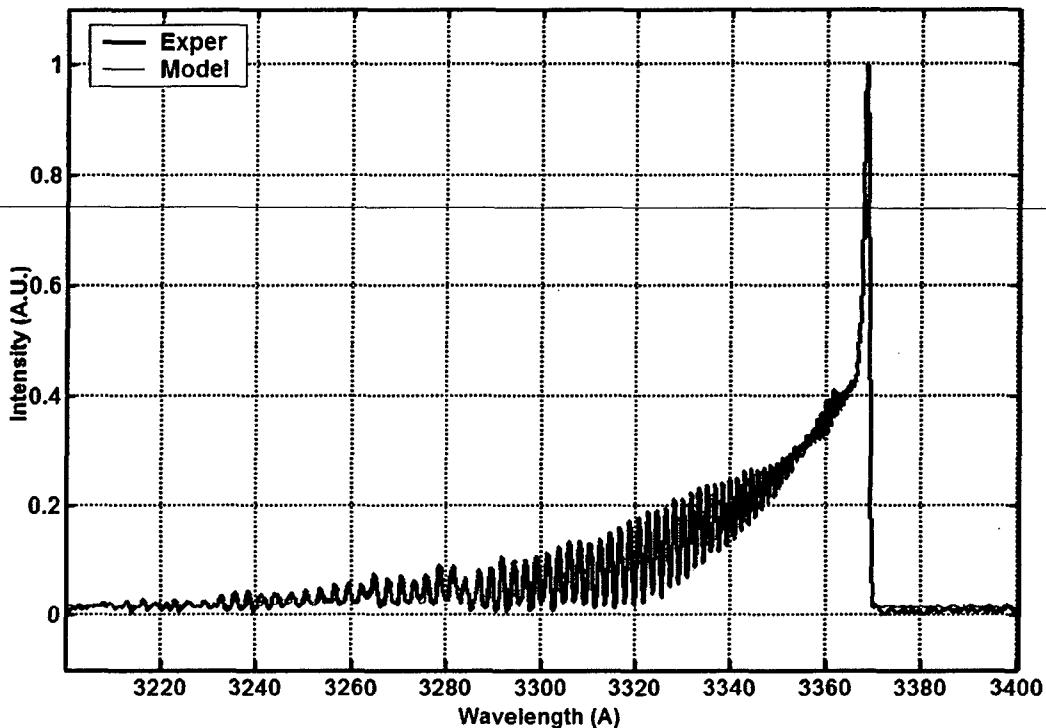

Fig. 7: SpecAir Code results for N_2 spectrum

The discrepancy between spatially averaged temperatures measured using different species (i.e. OH and N_2 in our case) can be explained particularly by the fact that measurements were conducted without spatial resolution and that maximum radiation of different radicals could come from different parts of the discharge with different temperatures. For example, the study of the gliding arc in air using optical methods[33] molecular spectra of OH and N_2^+ band heads at 306.3nm and 391.4nm were used respectively. They proposed the plasma column to be divided into two regions, one being the core in which the intensity of N_2^+ lines are observed and the other, the outer "flame" in which the radiation from OH is typical of the surrounding excited molecular region.

A spatially resolved spectrum was also obtained to determine N_2 rotational temperature in the cathode spot (CS) region (shown in Fig. 8) for 32mA, 37W conditions and was found to be ~ 1610K. A spatially resolved OH spectrum could not be obtained for the present experimental setup because the microscope used restricted our measurements to ~ 360nm and above. However, it is within the scope of our future work with the use of UV lenses.

Electric Field in MGA plasma arc

The snapshots in Fig. 8 show the gliding arc structure at different operating conditions (a) 35 mA (b) 102 mA and (c) 158 mA. For characterization of the plasma, we split the plasma into two zones namely – (1) Cathode Spot (CS) or Negative Glow (NG), and (2) Plasma Channel or Positive Column (PC). It was possible to notice that the CS width increased with current, thus keeping the current density nearly constant. This is typical for non-thermal atmospheric pressure glow discharge wherein the normal current density is, remains constant.[34, 35] However, it is important to resolve the electric field in these zones as it was expected to have a significant voltage drop in the CS.
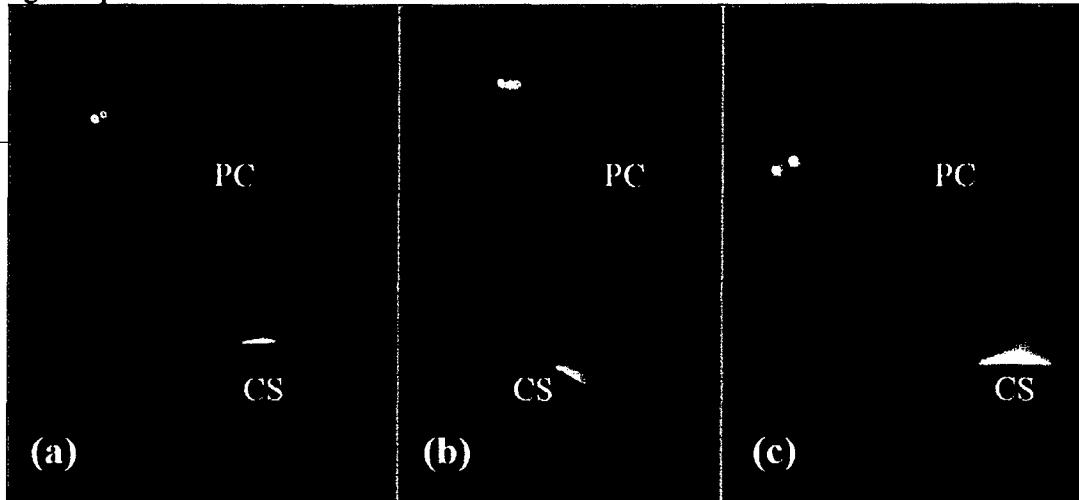

Fig. 8: Photographs taken by high speed camera capturing a single magnetically driven arc in motion. The two main regions of the discharge, namely the CS and PC can be clearly seen.

The typical value for voltage drops in the CS of glow discharges operating in air with iron electrodes ~ 260V.[35] Using visual quantification from high speed camera arc images, the approximate thickness of the cathode layer in our case (a) can be estimated ~ 0.03cm. Hence the electric field E ~ 8000 V/cm and the reduced electric field E/n ~ 175 Td (1 Td ~ 10^{-17} V.cm^2) using spatially resolved CS temperature T ~ 1600K.

It follows that the voltage drop in the PC ~ (1150 V – 260 V) ~ 890V. Again, imaging analysis yielded a length of the PC to be approximately 1.5 cm. Hence, the electric field E ~ 593V/cm and reduced electric field E/n ~ 17 Td – 23 Td, using spatially averaged T ~ 2200 K – 3000K.

The electric field can be used as a parameter for comparison of our discharge with non-thermal glow discharges where the reduced electric field values typically are in the range ~ 3 V/cm/Torr – 30 V/cm/Torr [35]. Using the calculated effective pressure parameters as discussed above, we estimate reduced electric field values for our conditions ~ 6 V/cm/Torr – 8 V/cm/Torr in the plasma column of the discharge, which is within the range of non-thermal discharges. Also, earlier investigations[45] for conventional GA reveal that it is possible to have a non-thermal regime at reduced electric fields as low as 2.4 V/cm/Torr.

Average Electron Energy Estimations

Using the parameters of the system, such as the geometry and assuming that the electric field is uniform through the PC, the reduced electric field range obtained was for the CS and the PC regions (computed earlier, above). A Boltzmann equation solver, BOLSIG[36] was employed to calculate the average electron energy versus the reduced electric field for air i.e. 80% N_2 and 20% O_2. The results obtained gave ~ 4.75 eV for the CS region and ~ 1.03 eV – 1.06 eV for the PC region at atmospheric pressure air conditions. These high electron temperature estimates

relative to the rotational gas temperature (energies ~ 0.13eV − 0.25eV) suggest the non-equilibrium nature of the MGA.

Current Density Estimations

From Fig. 8 it can be noted that there was a change in the width of the plasma column with an increase in current/power on the arc. Considering the arc to have a cylindrical structure, we assume that this width corresponded to the diameter of the gliding arc. By using these diameters and quantifying them based on the length per pixel from known dimensions, we obtain approximate cross-sectional areas of the arc column. Earlier, the frequency of arc rotation with current was measured. Using these results and the diameters/widths of the arc obtained by visual inspection and quantification at 1/8000 sec exposure time, we needed to incorporate a diametrical correction based on the frequency of arc rotation. These were approximated by the relation,

$$d = d_{meas} - \pi D f t$$

Where, d was the corrected diameter of the arc, d_{meas} − the measured diameter of the arc, D − the diametrical position in the device at which the measurement was taken, f − the frequency of arc rotation, and t − the exposure time. As mentioned earlier, for non-thermal characterization of plasma, we consider the arc to be divided into CS zone and PC (rest of the arc zone). Fig. 9 shows plasma current density (A/cm^2) results obtained by dividing the value of current with the cross-sectional area, calculated by assumptions as mentioned above. In low pressure non-thermal plasmas for air at room temperature and iron electrodes, typically current density is ~ 300µA/cm^2/Torr^2.[34, 35]
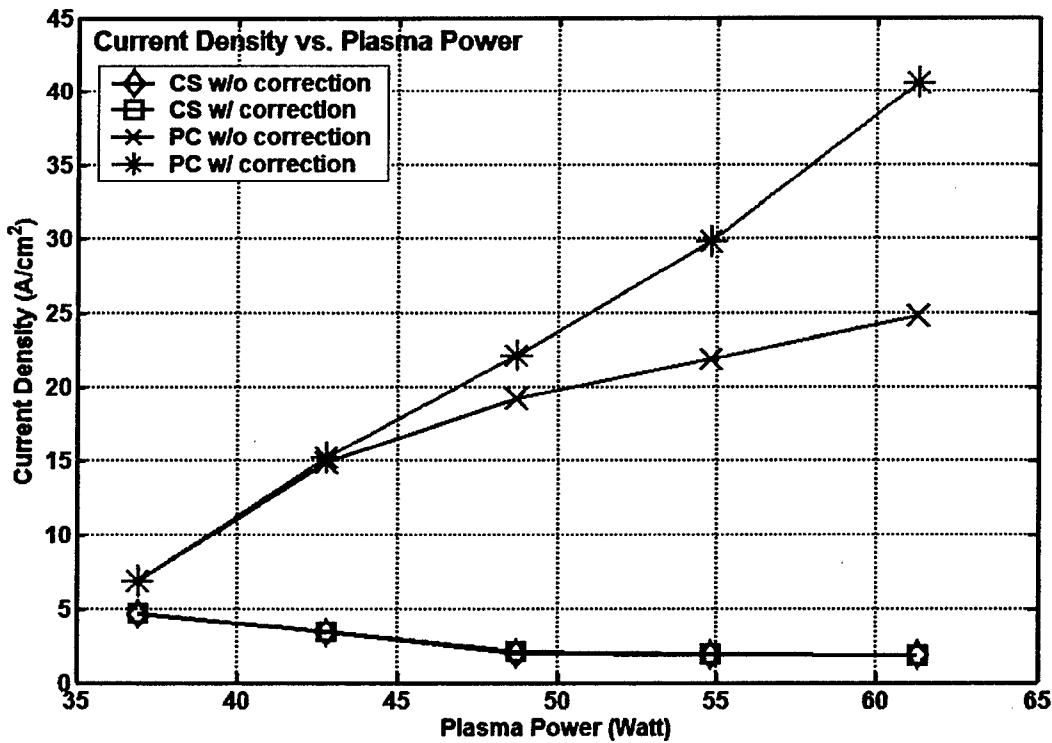
Fig. 9: Estimated MGA plasma current density in the CS and PC.

In low pressure non-thermal plasma, the ratio of electric field E to the pressure of operation p, i.e. E/p or ratio of electric field to the gas number density n, i.e. E/n, governs various plasma parameters. It is also sometimes known as the reduced electric field (REF). This scaling is applicable only if the gas temperature is that of the room, which is in the case of low pressure

non-thermal plasmas. However, in case of atmospheric non-thermal plasmas, the gas temperature mostly is higher than that of the room, in which case, a scaling needs to be incorporated. The REF parameter is scaled based on effective pressure, $P_{EFF} = P_{ATM} \cdot (T_{ROOM}/T_{ACTUAL})$. Where, P_{EFF} – effective pressure, P_{ATM} – atmospheric pressure, T_{ROOM} – room temperature and T_{ACTUAL} – is the gas temperature of plasma under consideration. The scaling is favorably formulated so as to preserve its initial values at room temperature.

Using this result for our conditions, i.e., $T \sim 1600$ K (from spatially resolved diagnostic results for CS zone) and scaling the pressure using number density, the effective pressure $P_{EFF} \sim 760 \text{Torr} \cdot (300K/1600K) \sim 141.6$ Torr and operating at 32 mA, 37 W conditions, we got a current density ~ 6 A/cm^2, which reasonably close to our experimentally estimated value in the CS zone ~ 4.7A/cm^2. Also, from spatially averaged rotational temperature values $T \sim 2200$ K – 3000 K, the current density estimate in the PC zone was ~ 6.9 A/cm^2. As the current was increased the temperature was expected to increase as well, which decreased the P_{EFF} value thereby decreasing the expected current density from literature estimations. This was observed in the CS data. However, in PC data an increase in current density was observed, which possibly could be compared to the property of contraction of non-thermal glow discharge with increase in current.

Electron Density Estimations

The electron density in the PC could approximately be estimated from current density, j and electron drift velocity, v_d, from the relation: $j/(e.v_d)$. From our estimations above, current density in the PC for $I = 32$mA is ~ 6.9A/cm^2. Using $T=2200$ K - 3000K, we applied a corresponding $E/n \sim 17$ Td - 23 Td into BOLSIG[36] to compute the electron drift velocity $\sim (2 - 3.7)$ x10^{7}cm/s, thereby the electron density $\sim (2.2 - 1.2)$ x10^{12} 1/cm^3 respectively, this gave an ionization degree $\sim (6.22 - 4.5)$ x10^{-7}. These results are reasonably consistent with earlier estimations in gliding arcs.[31, 32]

Thus, spectroscopic measurements of MGA temperature allowed to make discharge characterization that is necessary for coupling of the discharge properties with the ignition parameters.

"The Low Temperature Oxidation Chemistry of JP-8 and Its Surrogates at High Pressure"
(ARO, grant # DAAD19-03-1-0070)

In an effort to develop surrogate fuels for engine modeling and development, this project is examining the low and intermediate temperature oxidation chemistry of JP-8, potential JP-8 surrogates, and their components at elevated pressure. Experiments are being run in a pressurized flow reactor (PFR) and/or a single cylinder research engine. A fundamental understanding of the preignition chemistry of high molecular weight hydrocarbons, similar to, if not including, components of real fuels, is necessary to advance the development of fuel surrogates. This project is providing information necessary to determine the chemical reaction mechanisms of such hydrocarbons.

In prior work, we used gas chromatography coupled with a flame ionization detector or a mass spectrometer to measure the concentration of stable species produced during the low and intermediate temperature oxidation of JP-8 and suggested surrogate compounds in our reactor. Our goal with the spectrometer was to complement these measurements with spectroscopic measurements of radical species such as HO_2 and RO_2 using our recently developed Cavity Enhanced Magnetic Optical Rotation techniques.

The first step in this process is the assembly of a reliable room-temperature source of HO_2 to confirm and optimize our operating techniques. The system shown below was designed to create and observe HO_2 using cavity ringdown laser absorption spectroscopy (CRLAS). The system is entirely teflon to ensure safety from the reactive radicals involved. A microwave discharge is used to create a chlorine radical which then reacts with a 50% hydrogen peroxide solution to form HCl and the hydroperoxy radical. The spectroscopy of the mixture will be studied to ensure that the HO_2 radical has been created. FTIR spectroscopy with the CRLAS cell will be used to calibrate the hydroperoxy radical concentration.
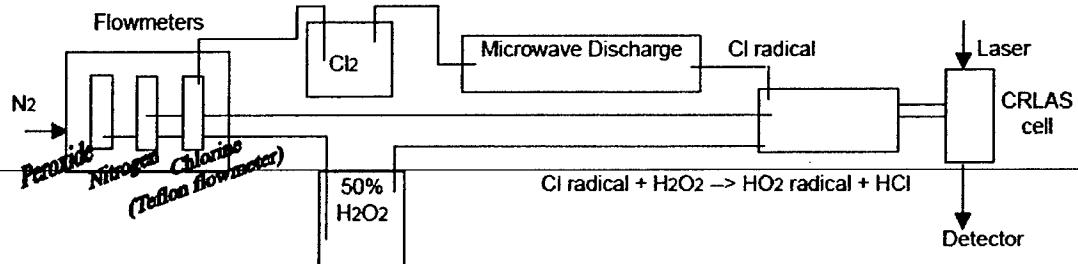
All HO_2 rovibrational lines will be mapped out and the source quantified by using published line strength information (Zahniser et al., 1989). Potential interference from other carrier species likely to exist in a reacting environment will be identified with low –pressure cells containing calibrated mixtures in addition to the USF-HITRAN database. After successful completion we can move on to additional verification studies and basic measurements of HO_2 in supersonic jet expansions. Ultimately, we plan to use the diagnostic to examine hydrocarbon reaction systems in our model reactors and engine system. Unfortunately, due to technical difficulties with our light source, a Nd: YAG/OPO laser system, current advancements have been delayed. Once our laser system is operational the spectrometer will be used in validating the HO_2 measurement and ultimately monitoring the concentration of this important combustion intermediate in our reactors.

"Atmospheric-Pressure Plasma Microdischarges for High-Rate Microfabrication" (NSF, grant # DMII-0423409).

Project Description:
    Atmospheric pressure non-thermal plasma discharges represent a lower cost alternative to current vacuum based plasma technologies used for micro-fabrication. This research involves experimental studies of micron-sized atmospheric plasma discharges using DC, RF, and microwave excitation. The plasmas are visually and electrically characterized for a variety of operating conditions and gases. Optical emission spectroscopy was used to measure the rotational, vibrational, and excitation temperatures. The micro-plasmas are being used for plasma enhanced chemical vapor deposition and sputter deposition of thin films. Mask-less micro-patterning has also been demonstrated. Deposited diamond like carbon films are being characterized by profilometry and Raman spectroscopy.

Applications of System:
    The Trivista spectrometer has been used to characterize several aspects of the microplasma. Using optical emission spectroscopy the spectrometer has been used for species identification. The system has the unique advantage of being able to provide high resolution spectra over a wide range of wavelengths. Figure 1 below shows the spectra and identified species in the 400 nm to 500 nm range for a RF discharge in air. Similar plots of species identification were

attained in the range of 200 nm to 900 nm. As seen the spectrometer provides resolved spectra of different vibrational and rotational transitions which in turn are used to determine the vibrational and rotational temperatures in the plasma.
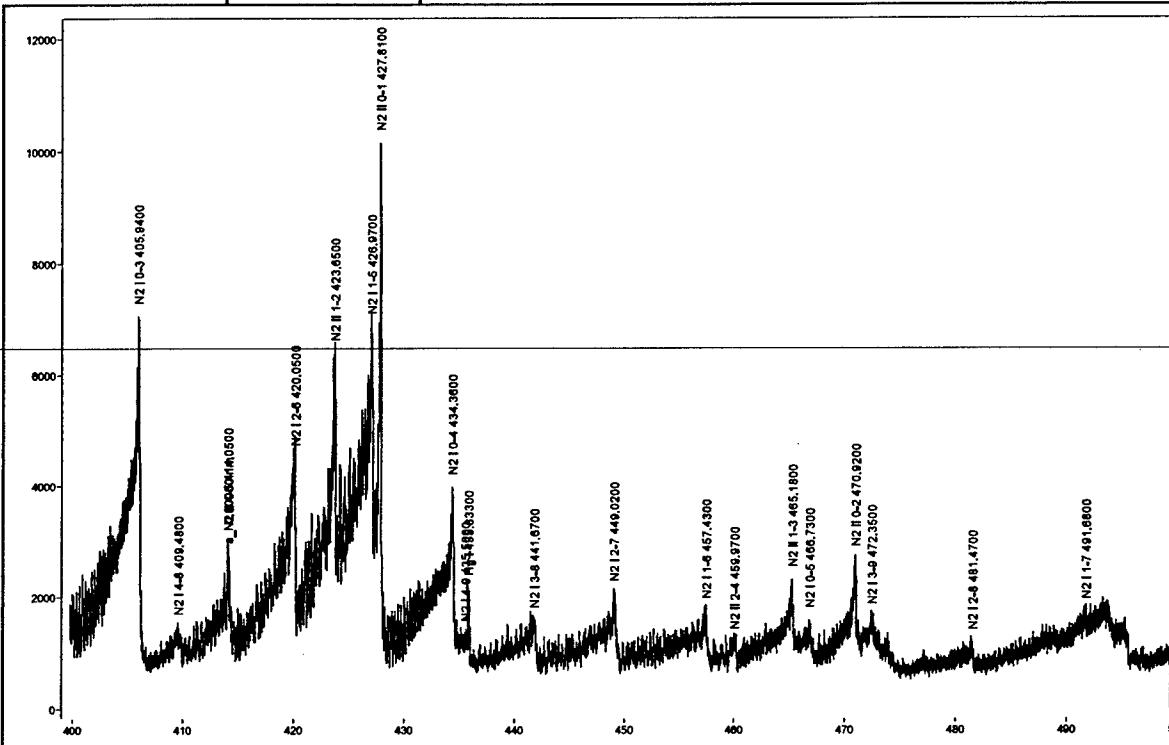

Fig. 1: measured spectra of a microplasma in air

The triple spectrometer was also investigated as a method to determine the velocity and energies of ions within the plasma. Ions as they cross the sheaths of a plasma are accelerated by significant electric field. Potential drops of as much as 300V might be expected. In vacuum conditions this leads to very high energy and high velocity ions. However in atmospheric pressure plasmas the ion energy is significantly reduced due to the presence of collisions within the sheath. There is some discussion as to what the ions energy are and whether drift velocity approximations, or ion momentum equations are necessary to determine the ion energy. Computational modeling has indicated ion energies as high as 5-6 eV; however other estimations of the ion energies indicate that it should not exceed 0.9 eV. The high resolution triple spectrometer system was used on these plasmas to help measure the ion velocity. As seen in figure 1 both ions and neutrals can be observed as emitting species in the plasma. The light emission from the ions will be Doppler shifted by an amount proportional to their velocities. The Doppler shifted wavelength will be $\lambda=\lambda_o(1+v/c)$. By using the triple spectrometer in an additive mode highly resolved spectra can be attained. The resolution of the spectrometer is about 0.02 nm which corresponds to a velocity of about 2700 m/s and N_2^+ ion energy of 1eV. Thus if the ion energy were greater than 1 eV a Doppler shift would be detectable. Thus far comparisons of spectra along and counter to the ion flow show no significant Doppler shifts. This experiment can be expanded to Helium plasmas discharges where due to the lighter ion masses the ion energy detection limit of the spectrometer will be 0.15 eV. Laser induced fluorescence techniques will also be implemented to get more accurate measurements of the ion and neutral velocities using the Doppler shift.

"Non-Thermal Open Air Plasma Discharge for Fast Blood Coagulation and Wound Sterilization" (DARPA, award # W81XWH-05-2-0068)

Fast coagulation of blood and sterilization of wounds is a problem faced by medical professionals on the battlefield as well as in hospitals. We are developing a new method for fast coagulation of blood and treatment of wounds that can be easily applied not only by a human operator, but by a remotely controlled machine. In this method a bleeding wound is exposed to a safe non-thermal atmospheric pressure plasma discharge recently developed at the Drexel Plasma Institute. Preliminary results on human blood and cadaver tissue indicate that the proposed plasma discharge coagulates blood quickly and sterilizes the wound without causing any damage to the tissue. While preliminary results are encouraging, a careful study of the proposed plasma based blood coagulation and wound treatment method is needed. This study and development of portable non-thermal open air plasma discharge for treatment of wounds is a goal of this project.

We developed a new non-thermal, room temperature plasma discharge operating in open air which is safe for treatment of living animal or human tissue (Fig. 1). This approach allows for novel treatment of biological and medical surfaces where no tissue damage is observed while some biological processes are initiated and/or catalyzed. Additionally we offer a model describing the influence of this plasma on concentrations of various ions in blood plasma. The principle of operation of the proposed plasma is similar to Dielectric Barrier Discharges (DBD) introduced by Siemens in the middle of 19th century. It occurs at atmospheric pressure in air when sufficiently high voltage of continuous waveform or pulses of short duration are applied between two insulated electrodes. The presence of the insulator between the electrodes prevents the build-up of high current. As a result, the discharge creates plasma without substantial heating of the gas, thus offering no medium limitations (i.e. need for lower pressure or noble gases). We found that it is possible to replace one of the electrodes by an object with high capacity for charge storage—a "floating electrode"(FE). Living tissue of animal or human body with its high water content and a relatively high dielectric constant has the required high capacity for charge storage and, therefore, can easily be employed as the FE of the DBD plasma. In this case the FE-DBD e-plasma is created in the gap between the living tissue and the other insulated electrode (Fig. 1). While the current in the gaseous discharge gap is mainly due to motion of charge carriers (electrons and ions), it continues mostly in the form of displacement current through the tissue. Moreover, most of the energy due to the e-plasma current is dissipated in the gap, just like in the case of a conventional DBD.

Treatment Target
- the Floating Electrode

FE-DBD Insulated Electrode
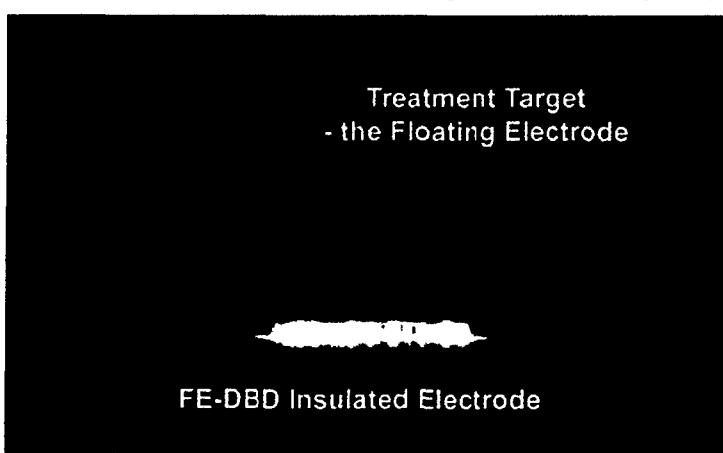
Fig. 1: Electrically safe non-thermal e-plasma for treatment of living tissue without causing damage

Thus, in the FE-DBD we obtain non-thermal plasma that remains at room temperature throughout the operation of the system while active species, radicals, ultraviolet radiation, and sub-millimeter scale temperature fluctuations offer "synergetic" effect in tissue treatment. Moreover, the plasma generated in this way can be applied directly to a living human tissue without thermal or chemical damage. In addition to sterilization of tissue, we demonstrated that

the FE-DBD plasma rapidly coagulates blood. Rather than a physical influence, we observe that e-plasma catalyses the natural blood coagulation processes as was confirmed by experimental data and a model based on plasma influence on ion concentrations in blood plasma.

We have developed several experimental systems to characterize the sterilization and coagulation processes as well as the discharge itself, because properties of the discharge have direct influence on the sterilization and coagulation. Depending on the discharge mode (AC or pulsed regime, discharge gap, humidity) plasma can be more or less uniform (Fig. 2, 3).
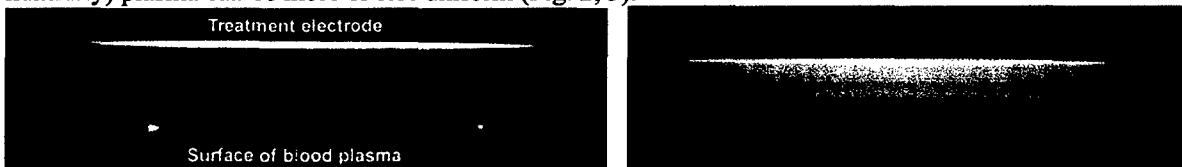

Fig. 2: Different modes of FE-DBD

Atmospheric pressure dielectric barrier discharge (DBD) have been used in many different applications during last decade. Atmospheric pressure and room temperature operating conditions are essential for processing of biologics and especially of living tissue of humans or animals. Under these conditions, however DBDs can form micro-channels that could heat up significantly over room temperature. Measuring temperature in these micro-channels is important to understand and improve the process of non-destructive treatment of living tissue.

Spectroscopic measurements were done using the system presented in Fig. 3. Figure 4 presents N_2 2nd positive system spectra for AC and pulsed FE-DBD. Transitions were modeled using SPECAIR; temperature determined by best-fit curve fit to model. It is possible to see that filaments in the pulsed discharge have significantly lower rotational temperature that usually corresponds to translational temperature. From this standpoint application of pulsed discharge for medical treatment is preferable.
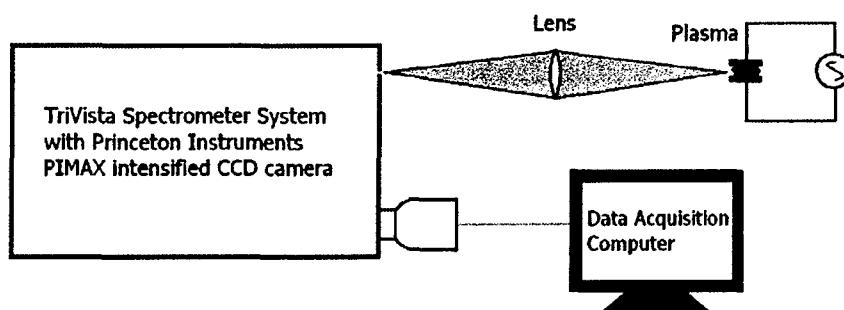
Fig. 3: Spectroscopic system used for spectroscopic characterization of FE-DBD

Next studies will involve spatial temperature distribution measurements of single Dielectric Barrier Discharge filament which will give more detailed understanding about possible temperature influence on the treated surface. Additionally OH concentration of various discharges will be measured with LIF technique in order to be able to tune plasma parameters finer.
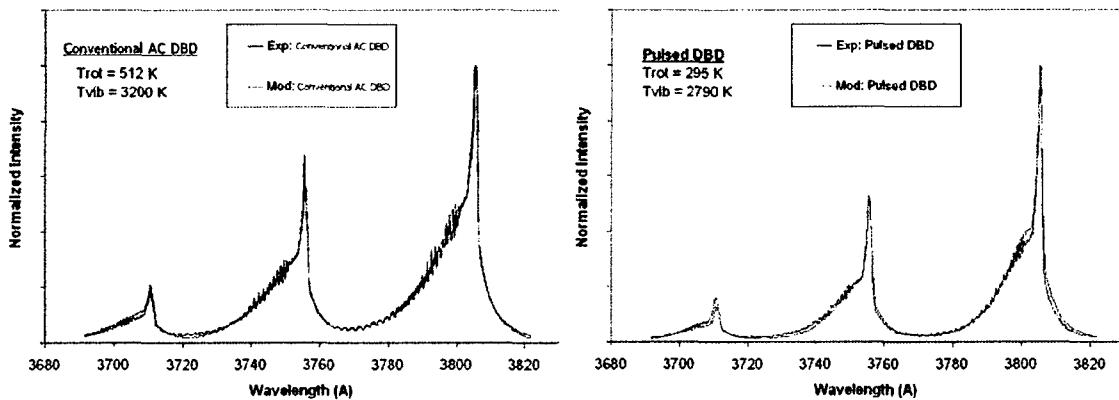
Fig. 4: Spectra for AC and pulsed FE-DBD

"Sterilization of Spacecraft Materials and Extraterrestrial Samples Using Atmospheric Pressure Cold Plasma" (NASA, grant # NNA05CS85G)

The central objective of this project is to elucidate the nature of the sterilization mechanism of cold plasma as it applies to the sterilization of spacecraft materials and extraterrestrial samples. Steam sterilization and dry heat sterilization in which the device is heated to a given temperature for a given duration in a prescribed atmosphere are currently the standard methods for these applications, however, material thermal sensitivity and requirements for energy and water make these methods unsuitable for sensitive components of flight hardware (for example, electronics) and space applications. Sterilization with chemical agents (ClO_2 or H_2O_2) damage treated substrates. Sterilization by cold ambient-air plasma avoids these problems and has been previously demonstrated successfully. Properties of non-equilibrium plasma are ideal for sterilization of spacecraft materials and dust samples. Energized particles, UV radiation and chemically active species can kill spores, bacteria and other microorganisms, at the same time, low kinetic energy (temperature) of the bulk gas in the cold plasma helps to preserve treated material from thermal degradation. Our research goal is to understand non-thermal mechanisms of sterilization caused by short lived plasma species that do not destroy spacecraft materials or the extraterrestrial samples.

From the standpoint of the mechanisms involved in plasma interaction with the treated surfaces, non-thermal plasma (NTP) sterilization systems can be divided onto three groups. NTP systems of the first group generate plasma which is then moved by the flow of the feed gas onto the surface to be treated. One of the advantages of this type of system is that the surface to be treated can be physically separated from the electrodes used to generate the plasma, which simplifies the design and operation of the device. However, the chemical species with the highest chemical activity are also those that have the shortest living time, which means that this flow of "decaying" plasma (sometimes called an "afterglow" or "plasma jet") exposes the surface predominantly to the flux of the lower activity, long-living chemical species. The lower concentration of ions that exist in afterglow are known to generate active chemical species and UV photons during recombination, but their concentration is much lower than in "active" plasma (plasma supported by electric field).

NTP systems of the second group are characterized by different types of discharges which periodically supply "active" plasma directly to the treated surface. These systems provide higher concentrations of active agents in pulsed mode, (short pulses, delivered hundreds or thousands of times per second), theoretically resulting in higher antimicrobial efficacy. The level of UV

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radiation which reaches the treated surface is higher for these technologies than for those of the first group, as there is essentially no intervening normal atmosphere between the plasma and the surface. One of the technical challenges with "active" plasma systems is that sensitive materials can be made to conduct electricity at sufficiently high voltages. Extended exposures of sensitive surfaces can result in electrical conduction through that surface, with the potential for damage, should the electrical conduction become concentrated in a single point. These NTP systems are therefore somewhat more challenging to build and operate than those of the first group. Gliding Arc (Fig. 1) is a typical system from the second group.

Fig. 1: Gliding arc plasma emitter for low temperature surface sterilization.

The third group of NTP systems places the surface to be sterilized instead of one electrode surface. In these systems, microorganisms are exposed to the broadest combination of active antimicrobial agents, at the highest possible intensity. Even more than with NTP technologies of the second group, the positioning of the sensitive surface to be treated must be carefully adjusted to avoid point-discharges, which can cause damaging levels of heat or electrical current to be concentrated at a single localized area. Although apparently the most promising of the types of NTP from an antimicrobial efficacy standpoint, these systems are among the most technically challenging. Typical example of the system from this third group is Dielectric Barrier Discharge (Fig. 2).

Each of these three general approaches (treatment with afterglow, treatment with active plasma, and treatment by "electrical contact") should be evaluated in the framework of our project to verify degrees of efficacy and applicability.

Fig. 2: Dielectric Barrier Discharge for low temperature surface sterilization.

Biological testing was started with two systems shown above. Figure 3 demonstrated SEM images of *Bacillus Subtilis* before and after DBD plasma treatment Comparison of Figure 3a (before DBD plasma treatment) and Figure 3b (after 10 min, 45C DBD plasma treatment) clearly shows that DBD plasma causes significant morphological changes and "physical" destruction of *Bacillus Subtilis* on the sterilization surface at the temperature 45C.

a)   b)

Fig. 3: SEM images of *Bacillus Subtilis* before (a) and after (b) DBD plasma treatment.
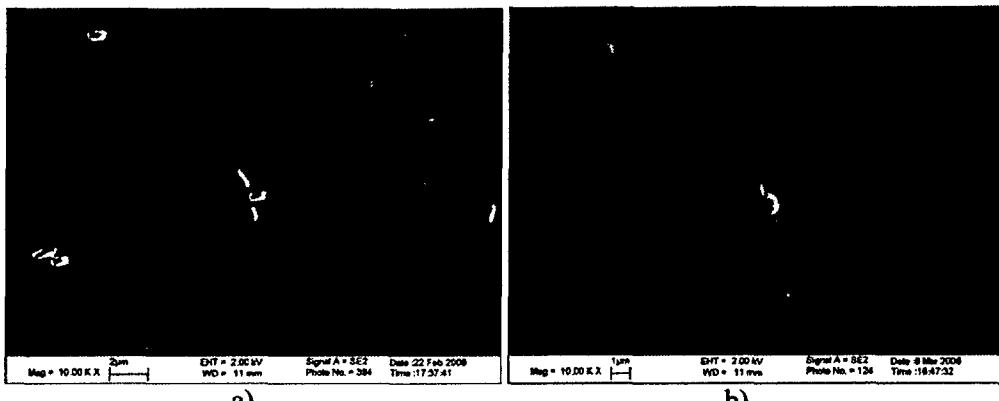
One of the future tasks of this project is quantitative analysis of the constituents of the plasma afterglow using GC/MS and spectroscopic measurements (emission in IR, visible and UV range), and LIF technique. For spectroscopic measurements we are going to use the system developed in the framework of the DURIP project.

Proposals and research tasks

In this chapter the submitted proposals and research tasks are listed and described that include use of the developed spectroscopic system.

"Fundamental Studies of the Non-Equilibrium Gliding Discharge in a Vortical Counter-Current Flowfield for Hydrogen Production" (NSF-DOE, rejected, will be resubmitted after modification)

Radical concentration measurements

To measure concentration field inside the reactor for key radicals in the plasma-catalytic process of hydrogen production, we are going to use LIF technique with space resolution during each shot (Fig. 1). In these experiments we are going to use Princeton Instruments Acton Research TriVista spectrometer with Intensified CCD Camera; Nd:YAG laser pumped Optical Parametric Oscillator (OPO), UV filters to monitor GA motion (reacting methane-air mixture should not emit radiation in UV range), and appropriate optics. OPO generator and CCD camera get "on" signal when both photomultipliers PMT-1 and PMT-2 generate signal simultaneously. Spectrometer slit will be oriented parallel to the OPO beam that is directed along the gliding arc motion. OPO excites molecules in the gliding arc plasma as well as in front of and behind the arc, and spectrometer provides special resoled measurements along the trajectory of GA motion. To avoid problems with light aberration in cylindrical quartz wall, preliminary adjustment of all devices onto *one point* will be made placing a photo-diode into *this point*. Also the available flat quartz lid of the reactor will be used in such measurements. Displacement of the reactor with the help of the second stepper motor (the first stepper motor will be used for arc elongation,) along its axis will allow measurements for different parts of the gliding arc without the optical system readjustment.
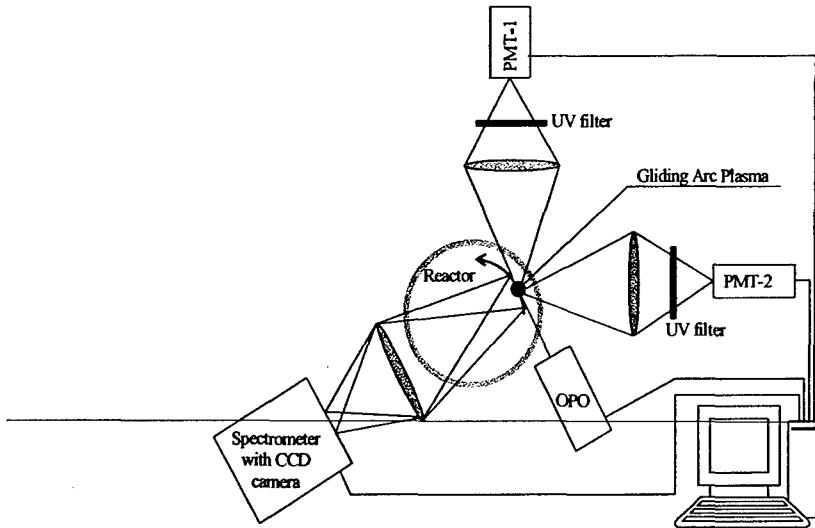
Fig. 1: Schematic for LIF spectroscopy diagnostics of the gliding arc discharge in vortical flow.

Measurement of spatial distribution of OH, CH, HO_2 and other radicals is important in developing an understanding of the low and intermediate temperature oxidation processes that take place during plasma catalyzed partial oxidation of methane and other hydrocarbons. Also the obtained results will be used for validation of the model proposed

"Dielectric Barrier Discharge Plasma Interaction with "Living Tissue" Floating Electrode" (NSF-DOE, rejected, will be resubmitted after modification)

Spectral investigations of radicals, atoms, exited particles and measurements of vibrational and rotational temperatures for FE-DBD plasma under various discharge conditions

High resolution spectrometer equipped with high speed CCD camera will be used for spectral investigation of FE-DBD plasma. Special software procedure developed earlier [55] will be used for measurement of vibrational and rotational gas temperatures from spectral data.

"Submerged Arc Plasma Disinfection of Water" (United States Israel Bi-national Science Foundation; rejected, will be resubmitted after modification)

Identification of species in the submerged arc (DPI)

A Trivista spectrometer system at DPI will be used to measure chemical species and radical production in the submerged arc. Spatially resolved measurements of the optical emission spectra (OES) of the discharge will be recorded in a range of 200 to 1800 nm. The Trivista spectrometer has sub-nanometer resolution (0.01 nm) which will show rotational sublevels and rotational temperatures within the discharges. A Princeton Instruments model 7467 CCD camera will be used to capture images of the discharges and a multi-channel plate (MCP) intensifier will be employed to compensate for a variety of lighting conditions. The obtained spectra will be compared to databases of known spectral lines to identify the species present.

Real time spectral absorbance measurement of transient species in treated water (DPI).

The feasibility of measuring the concentration of transient chemical species in the water in real time as a function of time from the end of the arc pulse, by absorption spectroscopy will be examined. For example, OH has rotational-electronic (0,0) transitions near 308 nm. The oscillator strength for this transition is $f=1.09\times10^{-3}$. An estimated absorption of 4% is estimated for an OH density-length product of $NL=10^{18}$ m^{-2}. If the absorption cannot be detected, at least

an upper bound on the *NL* product can be estimated. Measurements will be made at different lines of view through the water sample, whose distance from the electrode axis will be varied. An Abel transform will be used to obtain the radial concentration profiles at various times.

"Non-Thermal Atmospheric Pressure Electrical Discharge Plasma for Non-Surgical Treatment of Skin Diseases" (DARPA, Approved for funding)

System development

Depth of sterilization may depend on a number of plasma parameters. One of the most essential among them is the shape of the voltage pulse. This parameter is responsible for the extent of plasma uniformity and temperature in the micro-channels. In this task we will develop power supply capable of microsecond and possibly shorter pulses. We will also investigate the effect of electrode morphology on the rate and depth of sterilization. Electrodes constructed from insulated metal meshes will be used and plasma power and temperature parameters will be measured using colorimetric techniques, emission spectroscopy, and ultra-fast electrical measurements

"Energy Efficiency of Non-Equilibrium Plasma Ignition Control Above and Below the Autoignition Threshold" (AFOSR, under consideration)

Experimental Approach

In the framework of the proposed project we will perform reaction chain length measurements in fuel-oxidant jet flow reactor with excitation of separate reagents. We propose to separate particles of each particular type from the plasma generated in a separate volume and use these separated particles to initiate the chemical reactions in the premixed flow of fuel and oxidant. These experiments give us the possibility to analyze different mechanisms of chain propagation (radical, ionic, energetic) selectively initiated by different kinds of radicals, ionized and excited particles. As starting point we will investigate length of chains in H_2 - O_2 mixtures, and then the same approach will be used for hydrocarbon – air mixtures.

Let's consider how the proposed approach will be applied to the best studied combustible mixture H_2-O_2. Simple corona discharge in pure hydrogen (or/and oxygen in other series of experiments) (Fig. 1) will be used as a source of radicals and excited molecules only (when the grid (3) is grounded), or as source of ions only (when corona volume is elongated so that radical will recombine in volume and at the surface and grid (3) is under the floating potential); or as a source of ions, excited molecules and radicals. Ratio between numbers of radicals and excited molecules will be varied by pressure change, as at high pressure, collisional quenching of electronically-excited particles is much faster. Composition of the flow generated by the corona system will be carefully studied experimentally and by numerical simulation (see the chapter below), and then this flow will be injected to the main flow of combustible mixture (Fig. 3). Analysis of ion flux composition near the grid (4) will show what ions are stable and can work continuously as a catalyst in a combustible mixture. Our major attention should be paid to these ions. Injection of well-known radicals from the first corona generator into premixed flow will initiate chains, and injection of ions from the second corona generator should change the length of these chains. If ignition happens it means that the length of chains is enough to reach autoignition temperature, and this length will be calculated from the difference between initial temperature and autoignition temperature. In the cases without ignition, length of chains will be calculated after measurement of combustion product production (H_2O for H_2 – O_2 mixture) using spectroscopic measurements (IR-laser spectroscopy for H_2O).
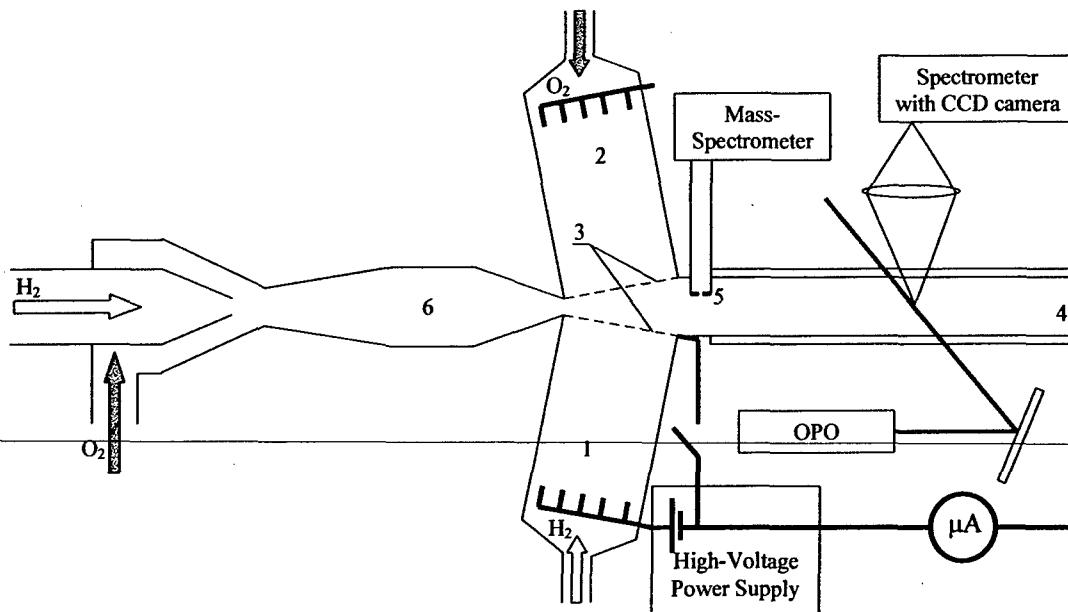
Fig. 1: Simplified scheme of the system for study of role of different plasma components in chain propagation support: (1) and (2) – corona plasma generators with high-voltage multiple needle electrodes; (3) and (4) – grids; (5) – pin-hole for mass-spectrometer sampling; (6) – generator of premixed flow.

So, control of the system temperature, pressure, composition of the injected and exhausted flows, will allow measurement of the length of chemical reaction chains and quantitative characterization of the role of different positive and negative ions, excited molecules and radicals in reduction of ignition temperature.

For reasons of safety, the first series of experiments in the system will operate with low-pressure (about 25 kPa) oxygen-hydrogen mixture. These experiments should demonstrate feasibility of our approach using the best studied combustible mixture. In the following series of more practical experiments with hydrocarbon-air mixtures the pressure will be close to the atmospheric one, as heavy gaseous hydrocarbons are safe in respect to possible explosion.

The composition of the mixture as well as combustion process will be controlled by means of emission/absorption spectroscopy. Spectroscopic investigations of $O_2(a^1\Delta)$, H_2O, OH, CH (LIF and PLIF measurements), O atoms (TALIF technique) will be supported by collaboration with Dr. Campbell Carter of Air Force Research Laboratory at Wright-Patterson Air Force Base. We also will measure an absolute H_2O yield in the reaction by IR-laser spectroscopy. To measure ion masses (and consequently to define their composition) we need to modify a mass spectrometer to extract ions from the system to the high vacuum and to accelerate them without use of additional ionization. For this part of work we are going to use experience of our colleagues from the Institute for Energy Problems of Chemical Physics of the Russian Academy of Sciences (Chernogolovka, Moscow Region).

Experimental research will include study of the cases with different corona polarities (influence of positive and negative ions), as well as with oxygen, hydrogen, and water vapor in the corona volume. Later the same technique can be used for gaseous hydrocarbons that are typically present in aviation fuel. If the role of ions in combustion is proved to be important, the next step should be study of the role of charged droplets and large clusters, which is especially important for higher hydrocarbons and other liquid fuels. Though the experiments with droplets and clusters are outside the scope of the proposed project, we suppose to consider this option during the experimental system design.

"Development of non-equilibrium plasma-flame kinetic mechanism and its validation using gliding arc integrated with counterflow burner" (AFOSR with Princeton University, under consideration)

<u>Experimental characterization of low-pressure non-equilibrium gliding arc for verification of the plasma kinetic mechanism</u>

The goal of this task is to characterize the gliding arc properties (electron temperature and gas temperatures) for a wide range of pressures and initial gas temperatures.

A spatially resolved temperature measurements using the N_2 spectrum, similar to that presented in our publication (Ombrello et al., 2006), will allow us to obtain a spatial distribution of the E/n parameter that is directly connected with the Electron Energy Distribution Function (EEDF). With a reasonable extent of simplification, EEDF can be presented by electron temperature T_e, which is a key parameter for plasmachemical kinetic simulations. Information about spatial distribution of E/n, gas temperature, T, and current measurements will give us the necessary information to calculate the electron concentration distribution (n_e).
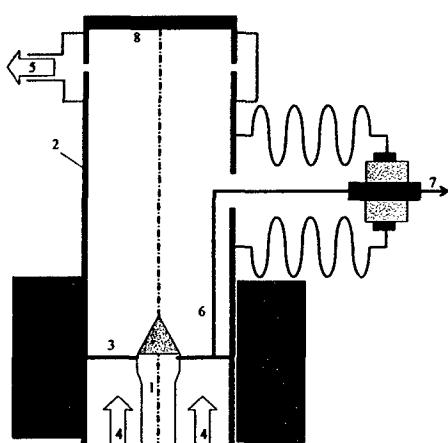
Also, additional information about the electron component of plasma will be obtained using the method of Langmuir probes that has been successfully applied to obtain reliable measurements of plasma properties in high pressure plasma arcs (Luhmann et al., 2002). Accurate electron temperature measurements can be made if the probe size is sufficiently small or moved through the plasma at a sufficient velocity (Clements and Smy, 1973).

Fig. 1: Magnetic Gliding Arc system with variable pressures: 1. anode, 2. cathode, 3. gliding arc plasma disc, 4. incoming gas flow, 5. exhaust flow to vacuum pump, 6. high voltage probe, 7. wire to oscilloscope, 8. optical window, 9. circular magnet.

Patents, Publications and Presentations

1. Gregory Fridman, Marie Peddinghaus, Halim Ayan, Alexander Fridman, Manjula Balasubramanian, Alexander Gutsol, Ari Brooks, Gary Friedman, "Blood Coagulation and Living Tissue Sterilization by Floating-Electrode Dielectric Barrier Discharge in Air" Plasma Chemistry and Plasma Processing, Vol. 26, 2006
2. Ombrello, T., Qin, X., Ju, Y., Gutsol, A., Fridman, A., Carter, C., "Combustion Enhancement via Stabilized Piecewise Nonequilibrium Gliding Arc Plasma Discharge", AIAA Journal 2006, 0001-1452 vol.44 no.1 (142-150)
3. Farouk T., Farouk, B., Staack, D., Gutsol, A., and Fridman, A., "Simulation of dc atmospheric pressure argon micro-glow discharge", Plasma Sources Science and Technology, V. 15, pp. 676-688 (2006).
4. T. Ombrello, X. Qin, Y. Ju, S. Gangoli, A. Gutsol and A. Fridman, "Non-Equilibrium Plasma Discharge: Characterization and Effect on Ignition", 44th AIAA Aerospace Sciences Meeting & Exhibit, AIAA-2006-1214, 9-12 January, 2006, Reno, Nevada.
5. Staack, D., Farouk, B., Gutsol, A., and Fridman, A., "Rotational and Vibrational Temperature Measurements of Atmospheric Pressure Normal Glow Plasma Discharges in Air, Nitrogen, Argon, and Helium", 3rd International Workshop on Microplasmas, Greifswald, Germany, May 9-11, 2006, IWM 2006 Proceedings, P. 102.

6. Staack, D., Farouk, B., Gutsol, A., and Fridman, A., "Rotational and Vibrational Temperature Measurements of Atmospheric Pressure Normal Glow Plasma Discharges in Air, Nitrogen, Argon, and Helium", 33rd IEEE International Conference on Plasma Science, June 4-8, 2006, Traverse City, Michigan, IEEE Conference Record - Abstracts, p. 156.

7. Gangoli S., Gutsol A., Fridman A., Ombrello, T., Qin, X., Ju, Y., "Characterization of Magnetically Stabilized Gliding Arc for Study of Flame Ignition and Stabilization", 33rd IEEE International Conference on Plasma Science, June 4-8, 2006, Traverse City, Michigan, IEEE Conference Record - Abstracts, p. 160.

8. Ayan, H., Fridman, G., Gutsol, A., Fridman, A., Friedman, G., "Homogeneous Discharge between Living Tissue and Insulated Electrode", 33rd IEEE International Conference on Plasma Science, June 4-8, 2006, Traverse City, Michigan, IEEE Conference Record - Abstracts, p. 267.

9. Fridman, G., Peddinghaus, M., Ayan, H., Fridman, A., Balasubramanian, M., Gutsol, A., Brooks, A., Friedman, G., "Blood Coagulation and Living Tissue Sterilization by Floating-Electrode Dielectric Barrier Discharge in Air", 33rd IEEE International Conference on Plasma Science, June 4-8, 2006, Traverse City, Michigan, IEEE Conference Record - Abstracts, p. 355.

10. Gutsol, A., "Warm" Non-Equilibrium Plasmas for Chemical Applications", Invited lecture at the Gordon Research Conference on Plasma Processing Science, July 16-21, 2006, Mount Holyoke College South Hadley, MA

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Issue date: April 14, 2005
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Item	Qty	Part Number and Description		Unit Price	Total Price
3	1	PI-MAX 1K UNIGEN 8019-0015	7467-0017 / 6050-0336	$37,420	$37,420
		PI Acton PI-MAX:1KHB Digital ICCD Camera System • e2v CCD 47-10 scientific grade 1, front-illuminated CCD, 1024 x 1024 pixels • 1:1 fiber-optic bonded 18-mm grade 1, Gen III extended blue image intensifier • Fiber-optic input, proprietary UV coating • 13 x 13 µm pixels (13.3 x 13.3 mm image area) < 5ns gating capable see data sheet for current specifications			
4	1	SPEC-MNT-PIMAX PI-MAX Spectroscopy mount	7389-0003	$210	$210
5	1	ST-133B/U-PTG-DUAL ICCD USB2 Controller with 16-bit 100-kHz & 16-bit 1-MHz digitizers, PTG	7513-0001	$11,000	$11,000
6	1	USB2-5M High speed communications port using USB2 protocol with 5 meter cable	6050-0494	$1,995	$1,995
7	1	WinSpec/32 Real-time acquisition, display, and data processing software for Microsoft Windows	4412-0048	$995	$995

All prices in U.S. dollars, F. O. B. Trenton, NJ
Sales tax will be added where appropriate
Payment Terms: Net 30 days, subject to credit approval
Shipping Method: Air Freight
Warranty: One year from date of shipment

Technical Inquires: Chris Meyers (609) 577-7442

Order Inquiries: Susan Carter (609) 587-9797

Est. shipping date: 120 days A.R.O. or sooner

Promotional Discount	-$14,888
TOTAL PRICE	**$129,712**

Authorized by:

Year 2 Warranty Special at time of purchase *(1/2 price of Silver Plan Coverage, below) $2,594.00 ☐check

* The RS Silver Service Package: The RS Silver Service Contract Package covers the customer's system 100%** labor plus parts with the exception of the (**CCD and/or Image Intensifier and software, which are not covered during the contract period).